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19 ABSTRACT (Continue on reverse if necessary and identify by block number) Chemical vapor deposition (CVD) processes promise inexpensive diamond optics and coatings with large dimensions. Near term optical applications include x-ray windows, membranes for x-ray lithography, and infrared windows and domes. CVD diamond, which is mainly polycrystalline, shows materials problems that include scattering due to large surface roughness, absorption due to defects, nondiamond carbon phases, and impurities, and poor diamond/substrate adhesion. Free carrier absorption in CVD diamond has also been reported. While diamond films less than 3 μm thick can be made transmissive in the visible and in the ultraviolet, at these wavelengths, thicker components scatter excessively and show absorption due to defects. Continuing research is improving the optical quality of CVD diamond. New polishing methods have yielded smooth surfaces in reasonable polishing times. Recent research holds promise for large optics made from single crystal diamond.														
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USE OF DIAMOND AS AN OPTICAL MATERIAL

Albert Feldman

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USE OF DIAMOND AS AN OPTICAL MATERIAL*

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INTRODUCTION

The high transmissivity of perfect diamond over extensive regions of the electromagnetic spectrum, in combination with diamond's great hardness, large abrasion resistance, high thermal conductivity, and chemical inertness make diamond a highly desirable optical material. Until recently, the high cost and small dimensions of optical quality diamond have limited the use of diamond to a few specialized optical applications; the best optical quality diamond, type IIa diamond, still comes from natural sources. However, new chemical vapor deposition (CVD) processes promise to make available inexpensive bulk diamond of large dimensions and thin film diamond over large areas, thus making possible the widespread use of diamond optics and optical coatings [1,2,3]. X-ray windows of CVD diamond are already commercially available [4]. Anticipated optical applications include infrared windows and domes, high power laser windows, and membranes for x-ray lithography [5]. Other possible applications include electroluminescent devices [6], lasers [7], and optical switches [8].

This article focuses on the status of CVD diamond as an optically transparent material. CVD diamond, which is mainly polycrystalline, exhibits several materials problems that limit its optical transmission, such as scattering due to large surface roughness and absorption due to defects, nondiamond carbon phases, and impurities. New polishing methods promise smooth surfaces in reasonable polishing times; however, work on surface figure, to my knowledge, has not yet been addressed. While intrinsic multiphonon processes limit the transmissivity of all diamond between 2.5 and 6.5 μm , CVD diamond usually

*This work was supported in part by the Office of Naval Research.

contains high densities of lattice defects causing normally forbidden single phonon absorption processes to occur between 7.5 to 12 μm . In addition, free carrier absorption in CVD diamond has also been reported. While diamond windows less than 10 μm thick can be made transmissive to visible and uv radiation, thicker components scatter excessively and show absorption due to defects.

Continuing research is improving the optical quality of CVD diamond. Recently, novel methods for producing single crystal (non-CVD methods) or near single crystal diamond have been reported. When fully developed, these methods could make available large optics of single crystal diamond.

High deposition temperature, large thermal expansion mismatch, and poor adhesion now limit the use of CVD diamond as a hard coating material. To overcome some of these difficulties, a method has been developed to place a diamond coating on ZnSe by bonding the coating to the substrate with a chalcogenide glass layer [9].

DISCUSSION

Deposition Methods. Several deposition methods are being used to deposit diamond by CVD. High quality diamond has been made by hot filament CVD [3], microwave plasma CVD [10], DC plasma torch [11], radio frequency plasma torch [12,13], microwave plasma torch [14], and oxy-acetylene torch [15]. Except for the oxy-acetylene torch method, the charge gas for producing diamond is hydrogen mixed with a hydrocarbon such as methane, acetylene, methyl alcohol, ethyl alcohol, or acetone [16]. The hydrogen fraction in the feed gas is almost always greater than 90 per cent. The quality of the diamond usually improves with increasing hydrogen fraction in the feed gas. It is also found that oxygen added to the charge gas improves the diamond quality [17]. Carbon monoxide mixed with hydrogen has also been used [18]. In the plasma torch methods, argon is sometimes used as a sheath gas. In the case of the oxy-acetylene torch, only acetylene and oxygen comprise the charge gas. In this case, deposition is carried out in the reducing part of oxygen-poor torch flame.

Substrate temperatures during deposition usually fall in the range 600 to 1000 $^{\circ}\text{C}$. Depositions have been done at temperatures below 400 $^{\circ}\text{C}$ [19]; however, the growth rates are low and the quality of the diamond, as determined by Raman spectroscopy, is poor. Recently, depositions by the oxy-

acetylene torch method at substrate temperatures up to 1600 °C have resulted in the growth of single crystal diamonds at high growth rates [20]. The method appears very promising for producing boule-sized single crystal diamonds of high optical quality.

A creative method for producing nearly single crystal diamond layers over large areas has been developed recently [21]. Selective area etching was used to produce an array of etch pits of a well defined pyramidal shape in a single crystal silicon substrate. A slurry containing single crystal diamond particles of a matching pyramidal shape was applied to the surface of the substrate and then removed. Diamond particles that had been left behind were embedded in each of the etch pits. These particles were crystallographically oriented to within several degrees. When the substrate was placed in a CVD reactor, the diamond particles acted as seeds for diamond growth, resulting in a continuous diamond film that was nearly a single crystal.

There have been two reports of single crystal diamond nucleation on single crystal copper substrates. Both methods rely on the lack of solubility of carbon in copper. In one experiment [22], carbon ions were implanted into a single crystal copper substrate at an elevated temperature. A transmission electron microscope diffraction pattern indicated that a single crystal diamond layer had formed on the surface of the copper.

In the other experiment, carbon ions were implanted into single crystal copper substrates at room temperature [23]. The surface of the specimen was then exposed to high power radiation from an excimer laser. The laser pulse energy was chosen to cause the surface of the copper substrate to melt and then to refreeze rapidly. During refreezing of the copper, the carbon atoms were expelled and forced to the surface, precipitating on the surface as a diamond layer.

A halogen-assisted CVD method has recently been discovered for producing diamond [24]; however, this research is in an early stage and has not yet resulted in good quality diamond films.

Optical Properties of CVD Diamond. Many of the projected optical applications of CVD diamond are based on the known intrinsic properties of diamond. The intrinsic absorption mechanisms of diamond are due to interband transitions of electrons across the fundamental electronic energy gap at 5.45 eV, and to multiphonon generation in the infrared between

2.5 and 6.5 μm . Generally, absorption due to 2-phonon and 3-phonon generation predominates; higher order phonon generation has significantly decreased probability. Absorption due to generation of single phonons is forbidden because of crystal symmetry. Thus, perfect diamond is transparent between 225 nm in the ultraviolet and 2.5 μm in the infrared, and from 6.5 μm in the infrared to zero frequency (DC). Because the absorption process in the infrared between 2.5 and 6.5 μm is of second order and higher, the absorption coefficients are small so that thin diamond films may transmit adequately for many infrared applications in this wavelength range.

CVD diamond is not yet of the quality of the best natural single crystals because of the high density of defects in the material [25]. Several types of defects are present. Lattice defects, such as grain boundaries, twin boundaries, stacking faults, and dislocations, break the crystal symmetry of the diamond lattice resulting in absorption due to generation of single phonons. Thus, absorption by single phonon creation in CVD diamond has been observed at wavelengths longer than 6.5 μm . Many of the principal features in the diamond phonon spectrum have been identified in these transmission spectra [25].

Impurities can produce electronic states within the electronic band gap or can lead to local vibrational modes resulting in unwanted absorption. Nitrogen is known to significantly limit the transmission of diamond in the ultraviolet and is responsible for absorption features in the infrared [26]. Hydrogen in combination with carbon produces a C-H stretch absorption feature in the infrared near 2800 cm^{-1} [25,27,28,29,30,31,32,33,34]. Other spectral features due to hydrogen have been observed in bulk diamond and may be present in CVD diamond [35].

CVD diamond usually contains nondiamond carbon phases that induce absorption especially in the visible and ultraviolet. The presence of nondiamond phases of carbon is usually detected by Raman spectroscopy. The Raman spectrum of pure diamond consists of a single peak located at 1332 cm^{-1} wavenumber shift. CVD diamond usually exhibits an additional broad peak near 1500 cm^{-1} that is attributed to a graphitic phase. The sizes of the two peaks (after subtraction for any luminescence background signal) is used as a qualitative measure of the diamond quality.

Recent experiments have shown that diamond can be made highly transmissive in the far infrared. Attenuation of the optical

signal was attributed both to free carrier absorption and to optical scatter [34].

Polishing CVD Diamond. CVD diamond films usually grow with surfaces that are undesirable for most optical application because of large surface roughness. Smooth diamond films can be made if the nucleation density is high; however, the thickness of such films is limited to several micrometers as the roughness tends to increase with increasing film thickness. However, in a recent paper, Wild et al. [36] have reported that diamond films grown with a <100> texture appear to grow smoother as the film thickness increases. Films of this type have been grown with thickness greater than 100 μm . Transmittance measurements indicate that these films show a higher optical transmittance than conventionally grown CVD diamond films.

Methods of polishing diamond films are being developed to produce smooth films. Because CVD diamond is polycrystalline and hard, it is very difficult to polish; polishing by conventional methods is very slow. Wang et al. [32] have polished CVD diamond films on a cast iron scaife heated to 350 °C. Six weeks of polishing were required to obtain a mirror-like surface. To increase the polishing rate a sample was annealed in an atmosphere of 0.01% oxygen in argon at 1000 °C for 4 hours; the film surface turned black. In this case, the time for polishing was reduced to one week. Polishing with potassium nitrate also increased the polishing rate; however, the specimen had to be carefully monitored to avoid destruction. Polishing decreased the peak-to-valley surface roughness from 1.2 μm to less than 0.1 μm .

Yoshikawa [37] has pioneered a thermochemical method for polishing diamond at high rates. In his method, a rotating polishing plate of iron or nickel is held at an elevated temperature inside an environmental chamber capable of supporting a vacuum. The CVD diamond surface is polished by holding it in contact with the rotating plate. In an atmosphere of hydrogen, iron produced the highest polishing rate and nickel produced nearly as high a polishing rate. No polishing action was observed with molybdenum or with cast iron plates and no polishing was observed at 700 °C or lower. At 750 °C and above, the polishing rate increased with increasing temperature. At 950 °C, the entire surface was polished after 20 min. The polishing rate also increased with applied pressure; however, excessively high pressures made the polishing process unstable. Increasing the lapping speed also increased the polishing rate. The average roughness, R_a ,

obtained on a 7 mm square specimen was 2.7 nm.

Frequently, the diamond surface is too rough for polishing directly. Yoshikawa has planed the surface of the specimen prior to polishing by irradiating the specimen with a Q-switched Nd-doped yttrium aluminum garnet (Nd:YAG) laser in one atmosphere of oxygen. A peak-to-valley roughness of 3 μm could be obtained by this process. Several authors have used variations of Yoshikawa's method to polish CVD diamond [38,39]

Protrusions that sometimes grow on the diamond surface must be removed prior to polishing. Harker et al. [38] have used reactive plasma etching to remove such protrusions. In order to etch only the protrusions and not the surrounding material, a nonreactive gold coating was applied to the entire surface. The protrusions were then exposed for reactive plasma etching with oxygen.

Another method of polishing CVD diamond by ion beam polishing has been developed [40]. The rough surface of a diamond specimen is spin coated with a mixture of photoresist and a Ti-silica emulsion to produce a plane surface. The surface is then etched with an oxygen ion beam; the angle of incidence is chosen to match the etching rate of the coating with the etching rate of diamond. The average root-mean-squared roughness on a 5 cm diameter film was 4.9 nm.

X-ray Window. Because of its low atomic number, carbon is highly transparent to x-ray radiation. The excellent mechanical properties of diamond make it useful as an ultrathin x-ray window for an energy dispersive x-ray fluorescence detector [4]. This is the first optical application of CVD diamond. In this application diamond is replacing beryllium which must be made considerably thicker to support vacuum. The optical transparency and mechanical stability of diamond also make CVD diamond a possible membrane material for x-ray masks. X-ray masks are used in the x-ray lithography of integrated circuits [5].

CONCLUSION

The superior properties of diamond make it a candidate for a number of optical applications. The most immediate application is the diamond x-ray window. The quality of CVD diamond for other applications, such as infrared transmissive elements and coatings, is continually improving but is not yet sufficient. Surface roughness is a major impediment to optical

applications. The ability to deposit smooth surfaces would make diamond considerably more attractive as an optical material. Polishing is being pursued as a means for making smooth CVD diamond surfaces. Diamond also has promise as a blue luminescent or laser material. Identifying and controlling the relevant luminescent defect centers will be needed to improve the quantum efficiencies of such devices.

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